IS 10391 (1982): Code of Practice for Chemical Cleaning of Boilers [CHD 13: Water Quality for Industrial Purposes]

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Indian Standard

CODE OF PRACTICE FOR CHEMICAL CLEANING OF BOILERS

(Fourth Reprint AUGUST 1999)

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BUREAU OF INDIAN STANDARDS
MANAK BHAVAN, 9 BAHADUR SHAH ZAFAR MARG
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AMENDMENT NO. 1 AUGUST 1986

TO

IS:10391-1982 CODE OF PRACTICE FOR CHEMICAL CLEANING OF BOILERS

(*Page 4, clause 1.1*) - Add the following note under clause 1.1:

'**Note** - This code is not applicable to the boilers of rivetted construction'.

(CDC 57)

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Indian Standard

CODE OF PRACTICE FOR CHEMICAL CLEANING OF BOILERS

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Indian Standard

CODE OF PRACTICE FOR CHEMICAL CLEANING OF BOILERS

0. FOREWORD

0.1 This Indian Standard was adopted by the Indian Standards Institution on 30 November 1982, after the draft finalized by the Boiler Water Sectional Committee had been approved by the Chemical Division Council.

0.2 Chemical cleaning of internal heating surfaces of boilers was first proposed where it was difficult or impossible to clean by other methods. Because of early experience with improper technique and limited knowledge of cleaning solvents, this method was at first subject to certain reservations. With progressive experience and development of materials and procedures, much of the earlier uncertainty concerning chemical cleaning has been resolved, and the process is now widely used with complete confidence as the quickest, cheapest and most efficient method of cleaning these surfaces in boilers of all sizes. It is, however, extremely important to use a procedure of known reliability under careful control.

0.3 Removal of pre-operational and operational deposits from the internal surfaces of steam generator tubing and other components has become an increasingly important maintenance problem in modern equipment. Because mechanical cleaning is virtually impossible in modern boilers, the effective application of chemical cleaning solvents has become a necessary tool of the power plant operator. Selection of a solvent system and procedure should be based primarily on three criteria namely, cleaning effectiveness, economics and base-metal attack.

0.4 The importance of maintaining clean heat-transfer surfaces in boilers has been recognized for many years. Tube cleanliness becomes increasingly more important in higher pressure boilers for two obvious reasons: (a) higher operating temperatures reduce the temperature elevation that can be tolerated without exceeding metal creep limits, and (b) higher temperatures are conducive to chemical corrosion which can proceed beneath internal deposits where soluble boiler-water constituents can concentrate.

0.5 The procedure outlined in this standard is recommended with the understanding that the user will depend upon competent chemical supervision of the cleaning operations as also expert advice of consultants on boiler-water and scale problems.

0.6 In the preparation of this standard, assistance has been derived from the following publications:

BS 2486 : 1978 Recommendations for treatment of water for land boilers British Standards Institution.

ATWOOD (KL) and HALE (GL). A method for determining need for chemical cleaning of high pressure boilers. Proceedings of American Power Conference, 20-22 April 1971.

Steam and its use. 1967. Babcock & Wilcox Ltd, USA.

Water chemistry for high pressure boilers — Reference manual. 1978. Bharat Heavy Electricals Ltd, Tiruchirapalli.

1. SCOPE

1.1 This standard lays down guidelines for chemical cleaning of boilers.

2. PURPOSE OF CHEMICAL CLEANING

2.1 The primary reasons for chemical cleaning of boilers are to prevent tube failures and improve unit availability. Tube failures in low-pressure boilers are normally the results of creep which occurs when internal deposits produce excessive metal temperature. A relatively smaller quantity of deposit creates difficulties in high-pressure boilers. Caustic corrosion and hydrogen damage, which occur only in the presence of deposits, may cause tube failures at temperatures well below the creep limit. Deposits originating both from fabrication and during operation should be considered potential problems.

2.2 All the pressure parts of a steam generator may be subjected to heat treatment of some sort during fabrication or erection — during forming operations, stress relief, welding or bending. Whenever carbon low alloy steels are subjected to high temperatures in the presence of air, oxidation occurs; the oxide produced is known as mill scale. Mill scale on boiler tubing is normally very thin with the exception of areas near welds and bends. Even where mill scale is initially uniform its brittleness upon cooling may produce flaking. The resulting non-uniform surface is undesirable from the standpoint of corrosion susceptibility. During operation mill scale is readily eroded from the steam-generating surfaces and may subsequently be redeposited in critical areas. Preoperational acid cleaning removes mill scale and serves to remove atmospheric rust which inevitably accumulates to some degree during erection.

2.3 After a boiler is placed into service, numerous solid constituents may enter the unit with the feed water, and some portion of the insolubles can be expected to deposit on boiler surfaces. If not removed, these deposits accumulate over a period of time to the point where they cause difficulty. Control of preboiler corrosion can minimize the quantity of these materials; however, complete freedom from deposition is not possible in a high-pressure system. The need for occasional chemical cleaning during the life of a boiler has become a recognized fact and should be accepted as a routine maintenance practice. A frequency of service cleaning of every 3 to 4 years is recommended. This frequency should be increased if individual unit operating history dictates.

2.4 Deposits of calcium and magnesium may be present in units that experience condenser leakage. This is the exception, and operational utility cleanings are directed primarily towards removal of preboiler corrosion products. For this reason, similar procedures are employed for both post-operational and preoperational utility-boiler cleaning. Iron oxide removal is the major requirement in both cases. Preoperational cleaning should be preceded by an alkaline boil out to facilitate removal of silica and organic preservatives, while operational cleaning should, in most plant cycles, eliminate copper either simultaneously or in a separate step.

3. ADVANTAGES OF CHEMICAL CLEANING

3.1 Compared with the removal of scale and deposits from internal heating surfaces by mechanical means, important advantages of chemical cleaning as are follows:

a) Outage time is reduced.

b) Extensive dismantling of the unit is not required. The solvents can be introduced and discharged through existing and some extra connections. The only dismantling necessary is the removal of drum manhole covers and a few header handhole closures for inspection. In some cases it may be necessary to remove drum internals also depending on the quantity of deposits and the technique involved.

c) The total cost is less.

d) Inaccessible areas, such as small tubes, short-radius tube bends and irregular surfaces, are readily cleaned since the cleaning medium has access to all locations reached by steam or water. The result is a better cleaning job. The more thorough removal of scale reduces the rate of subsequent formation of deposits.

e) New equipment can be designed with complete flexibility and simplicity for best economy, efficiency and overall cost, since no compromise is required to provide accessibility for cleaning.

4. GENERAL METHODS

4.0 Two methods of chemical cleaning have been recommended, namely;

a) continuous circulation of the solvent through the parts to be cleaned, and

b) filling the unit with solvent and allowing it to soak for a prescribed length of time.

4.1 Circulation Method — In the circulation method, after filling the unit the solvent is recirculated until cleaning is complete. Samples of the return solvent are tested periodically during the cleaning. Cleaning is considered complete when the acid strength of the returned solvent reaches equilibrium, indicating that no further reaction with the deposits is taking place. Compared with the soaking method, since a solvent of lower strength may be used and the solvent is drained from the unit as soon as the reaction is complete, the possibility of damage to the surface cleaned is less in using the circulation method. Also, the cost of chemicals is somewhat less because of the control testing of solvent strength.

4.1.1 The circulation method is particularly suitable for cleaning forced-flow boilers, superheaters and economizers with positive liquid flow paths to ensure circulation of the solvent through all parts of the unit. A complete cleaning job cannot be anticipated by this method unless the solvent reaches and passes through every circuit of the unit.

4.1.2 Cleaning by the circulation method is aided mechanically by the constant change of solvent which flows over the deposits on the surface This feature is of particular advantage when foaming may occur, as with deposits of a calcareous nature. The continuously circulating solvent also carries disintegrated undissolved materials through the unit. This is especially helpful in preventing clogging of small tubes. Circulation also limits the concentration of ferric chloride, which itself is an active corrodant against which inhibitor gives very little protection.

4.2 Soaking Method — In cleaning by the soaking method, after filling with the solvent the unit is allowed to soak for a period of 4 to 8 hours depending upon deposit condition. To ensure complete removal of deposits, the acid strength of the solvent should be somewhat greater than that required by the actual conditions since, unlike the circulation method, control testing during the course of the cleaning is not conclusive, because samples of solvent drawn from convenient locations may not truly represent conditions in all parts of the unit.

4.2.1 The soaking method is preferable for cleaning units where definite liquid distribution to all circuits by the circulation method is not possible without the use of many chemical inlet connections or where circulation

through all circuits at an appreciable rate cannot be assured except by using a circulating pump of impractical size. These conditions may exist in large natural-circulation units with complex furnace-wall cooling systems.

4.2.2 Advantages of the soaking method over the circulation method are:

a) Simplicity of piping connections,

b) Assurance that all parts are reached by a solvent of adequate acid strength,

c) And less chemical supervision during the cleaning operation.

5. SOLVENTS, DEPOSITS AND INHIBITORS

5.1 Solvents — For the removal of boiler deposits, various solvents including acids and a number of alkaline compounds have been recommended in Table 1.

In general, the use of alkaline compounds requires considerable time and temperature, and while the attack on steel is non existent or negligible, the surface is not as clean as with the acid solvents.

5.1.1 On the basis of cost, availability, suitable inhibitors, and rapid removal of deposits, hydrochloric and sulphuric acids are the most practical cleaning agents. Of these two, hydrochloric acid costs less, is more effective in dissolving a greater number of the usual constituents of boiler deposits, and is more readily available and inhibited. In contrast to the reactions of sulphuric acid which produces relatively insoluble sulphates, the reactions of hydrochloric acid form readily soluble chlorides.

5.1.2 Pressure parts of alloy steels, particularly those high in chromium, should not be cleaned with acid solvents without competent chemical advice on the type of acid and inhibitor and the conditions of application in each specific case.

5.1.3 For deposits of certain characteristics, the solvent may require additional reagents, such as intensifiers to react with certain constituents or wetting reagents to promote solvent penetration of the deposit. The need for these auxiliary chemicals should be decided by the chemist controlling the specific cleaning operation.

5.2 Deposits — Preoperational scale deposits and contaminants on the internal heating surface of a boiler unit include mill scale, weld scale, corrosion products, oil, grease, dust and dirt, temporary protective coatings

TABLE 1 TYPES OF DEPOSITS AND RECOMMENDED SOLVENTS FOR CLEANING

(*Clause* 5.1)

Sl No.	Cleaning Solutions	Carbonate	Phosphate	Sulphate	Silicate	Copper and Its Oxides	Mill Scale	Magnetite	Petroleum Oils and Grease	Vegetable Oils and Fats
(1)	(2)	(3)	(4)	(5)	(6)	(7)	(8)	(9)	(10)	(11)
i)	Hydrochloric acid	S	S				S	S		
ii)	Hydrochloric acid + acidic fluoride	S	S		S		S	S		
iii)	Sulphuric acid						S	S		
iv)	Sulphamic acid	S								
v)	Tetrasodium salt of EDTA	S	S	S						
vi)	Tetra-ammonium salt of EDTA	S	S	S		S	S	S		
vii)	Citric acid						S	S		
viii)	Ammoninm citrate pH 3 to 4						S	S		
ix)	Ammonium citrate pH 9·5 with oxidizing agent					S				
x)	Formic acid + hydroxy acetic acid	S	S				S	S		
xi)	Sodium hydroxide + sodium phosphate				A				A	S
xii)	Sodium carbonate + tri-sodium phosphate								S	S
xiii)	Organic solvents A — effective for light deposits S — effective								S	

NOTE — With the majority of the solvents an effective corrosion inhibitor is essential.

and other contaminants remaining after fabrication. Scale deposits formed on the internal heating surfaces of a boiler unit come from water. In general, the constituents belong to one or more of the following groups:

a) Oxides of iron and copper;

b) Carbonates, phosphates and sulphates of calcium and magnesium; and

c) Silica and silicates. The deposits may also contain various amounts of oil, which are sometimes appreciable in boilers on return to service.

5.2.1 For the most efficient and economical scale removal, the deposit should be analysed and that acid should be selected for the solvent which will most effectively react with a deposit of this type. This deposit is usually a mixture of several compounds, and the acid that will best dissolve the different major constituents is generally selected as the solvent since the dissolution of the soluble components will free any insoluble compounds present which can then be removed by flushing.

5.2.2 To avoid possible redeposition of other iron compounds, hydrochloric acid is usually preferred in removing iron oxide, even though sulphuric acid is theoretically the better solvent. Hydrochloric acid is more effective than sulphuric acid in dissolving calcium and magnesium salts. Neither has any effect on calcium sulphate, although scales containing up to 75 percent of this compound have been successfully removed by dissolving the soluble constituents.

5.2.3 No universal recommendation has been made for silica or silicates because some silicates react more readily with hydrochloric acid, some with sulphuric acid, and still others with a combination of strong alkalis and heat. A preboil for several hours with a 0·5 to 1 percent soda ash solution before acid cleaning has been found effective in changing the silica scale to a more acid soluble form. In some instances, to remove silica scale hydrofluoric acid has been included in the solvent. Except under expert chemical advice and supervision, this procedure is not recommended.

5.2.4 Small amounts of oil in the deposit are removed when the general deposit is dissolved and disintegrated. For higher percentages of oil contamination it may be necessary to use a wetting agent in the solvent to promote penetration of the deposit. If the deposit is predominantly oil or grease, boiling out with alkaline compounds should precede the acid cleaning. In new or repaired boilers where water-soluble lubricants have been used in tube expanding, boiling out with alkaline compounds may be omitted.

5.3 Inhibitors — The following equations represent the reactions of acids (in this instance, hydrochloric) with constituents of boiler deposits:

$$Fe_3O_4 + 8\,HCl = 2\,FeCl_3 + FeCl_2 + 4\,H_2O$$
$$CaCO_3 + 2\,HCl = CaCl_2 + H_2O + CO_2$$
$$Fe_2O_3 + 6\,HCl = 2\,FeCl_3 + 3\,H_2O$$

At the same time, however, the acids will also react with and waste away the metal of the boiler, as represented by the equation:

$$Fe + 2\,HCl = FeCl_2 + H_2$$

unless means are provided to prevent or retard this reaction without, of course, affecting the attack on the deposits. For this purpose arsenic compounds, barium salts, starch, quinoline, pyridine or other products have to be added to the acid solvent as inhibitors to prevent or materially reduce this reaction with the metal. A number of commercial inhibitors are sold under brand names, and still others are made and used by companies furnishing complete acid-cleaning service, including the prepared solvents. If cleaning is done by the operator's own organization rather than by a company specializing in this work, the use of commercial inhibitors offered by reputable chemical concerns is recommended.

5.3.1 The following four such commercial inhibitors are available for this purpose:

a) *Rodine 213* (*ordinary*) — used with hydrochloric acid at temperatures up to 40°C for inhibiting copper, brass, iron and steel;

b) *Rodine 213* (*special*) — used with hydrochloric acid at temperatures up to 80°C for inhibiting copper brass, iron and steel;

c) *Rodine 115* — used with sulphamic, citric, tartaric and oxalic acids for inhibiting copper, brass, mild steel and stainless steel; and

d) *Rodine 92* — used with sulphuric, phosphoric and citric acids.

Other inhibitors which satisfy the requirements for both inhibitor efficiency and toxicity aspects may also be used. Details regarding their concentrations, inhibiting efficiency to particular metal at definite temperature and toxic effects are generally obtained from the manufacturer. However, it is desirable to verify by conducting tests before actual use. The approved inhibitors should be present under acid conditions at all times to ensure that they are fully dissolved and not

subsequently precipitated. If these are recommended for use with austenitic steel surfaces, chloride content in such inhibitors should be practically nil so as not to impart more than 0·1 mg/l chloride in the solution used for cleaning.

NOTE — Whenever a new batch of inhibitors is used, its effectiveness should be tested before use. Pre-inhibited solutions should also be tested before use as the inhibiting efficiency may go down on storage.

5.3.2 The effectiveness of acids in attacking boiler deposits as well as steel increases rapidly with rise in temperature. However, the inhibiting effect decreases as the temperature rises, and at a certain temperature, which is likely to vary for each inhibitor, the inhibitor may be decomposed into compounds devoid of inhibiting properties. It should also be noted that not all inhibitors are effective with all acids.

5.3.3 Important points in the use of inhibited acid solvent are summarised as follows:

a) Selection of the acid and auxiliary chemicals should depend upon the type of deposit to be removed,

b) The inhibitor should be suitable for the acid selected,

c) The temperature of the solvent should not exceed that at which the effectiveness of the inhibitor will be reduced or destroyed, and

d) The limiting temperatures for the use of the inhibitor should be obtained from the supplier or manufacturer.

6. DETERMINATION OF SOLVENT CONDITIONS

6.1 Deposit Samples — The preferred type of deposit sample is a small section of tube with adhering deposit. In any case a sample of the deposit, down to the bare metal, representing the worst condition in the unit, should be obtained. Selection of the acid and auxiliary chemicals is then based on an analysis of the sample by chemicals or X-ray diffraction, or by other methods. After selection of the acid for the solvent and the inhibitor suitable for this acid, it is necessary, to ensure complete removal of the deposits, to determine the acid strength of the solvent, the solvent temperature, and the length of time required for the cleaning process.

6.2 Acid Strength — Technically, the acid strength should be in proportion to the amount of deposit in the unit to be cleaned. However, since the determination of the amount of deposit cannot be much more than rough approximation and the general average of conditions

observed falls within a fairly narrow range, the acid strength of the solvent is usually set satisfactorily by experience. It is, therefore, recommended that the acid strength of the solvent, as fed to the unit should be:

a) three to five percent when the unit is cleaned by the continuous circulation method, and

b) five to six percent when the unit is cleaned by the soaking method.

6.3 Solvent Temperature — To aid in the acid attack on the deposits, the temperature of the solvent should be as high as possible without seriously reducing the effectiveness of the inhibitor. Most commercial inhibitors break down or lose their effectiveness entirely above 70°C. As the temperature rises above 60°C the corrosion rate increases. It is, therefore, recommended that the temperature be limited to 60°C unless a higher temperature can be tolerated in a particular unit and with an inhibitor safe at the higher temperature.

NOTE — Before injecting any chemical, the boiler may be fired slowly with low heat if steam heating facilities are not available, to raise the temperature of water. However, this should be done carefully as the temperature may not be uniform at all places and may also overshoot the recommended limits. No firing should be done during chemical circulation.

6.3.1 Care should be taken to inspect the unit thoroughly after the first cleaning and after each subsequent cleaning. In this way the actual corrosion rate can be determined, and it can be decided either to continue cleaning at this temperature, if the rate is not damaging, or to reduce the temperature for subsequent cleanings. In making up the solvent to the required strength, it should be heated by warm water to 60°C or to the temperature decided upon after all conditions are known. The temperature of the solvent should not be raised by firing the unit. Circulation rate should be between 0·3 m/s and 1·0 m/s in the circulation method for effective cleaning.

6.4 Cleaning Time — When cleaning by the circulation method, completion of the process is determined by analysing samples of the returned solvent for acid strength and noting when the acidity reaches equilibrium. In using the soaking method, the length of time the solvent is to be retained in the unit should be decided upon beforehand. While the types and quantity of the deposit and laboratory determination of the reaction rate of the solvent on samples of the deposit should apply in setting the length of time, a practical guide in determining the solvent retention time for the first few cleanings, using the acid strength noted above, is given below:

a) For moderate thickness of relatively soft sludges or for conditions that would otherwise require turbining at not less than yearly intervals, the solvent retention time should be a minimum of 4 hours.

b) For thin coatings (eggshell thickness) of relatively hard scale formation, the solvent retention time should be 6 hours.

c) For deposits like those resulting from a severe upset in the feed-water-conditioning system, the solvent retention time should be a maximum of 8 hours.

6.4.1 The periods noted are for the actual retention of the solvent in the unit. The surfaces are, of course, also wholly and partially subjected to the solvent during the time of filling and draining. In establishing permanent schedules, the periods recommended should be extended or shortened on the basis of the results achieved for specific conditions during the first few cleanings, as indicated by visual inspections; test turbining of selected tubes, before and after cleaning, in areas typical of the worst deposit conditions; or examination of sample sections of tubing cut out of the unit.

7. PREPARATION FOR CLEANING

7.1 Heat Transfer Equipment — All parts not to be cleaned should be isolated from the rest of the unit. To do this, connections to these parts may be removed and blanked off or plugged with wood or rubber. Where arrangements permit, parts of the unit can be isolated by completely filling with hydrazine-dosed water. Bronze or brass parts should be removed or replaced temporarily with steel ones. All valves should be of steel or steel alloy. Gauge and meter connections should be closed or removed from the unit. All parts of the unit not otherwise protected by blanking off or by flooding with water should be covered by the inhibited solvent. Vents should be provided wherever vapours might be pocketed, since acid vapours from the cleaning solution do not retain the inhibitor.

7.2 Cleaning Equipment — Continuous circulation requires sufficient inlet connections to ensure distribution and to promote circulation through all circuits. It also requires a return line to the sump tank from the highest point of the unit or from the end of a continuous-tube heat transfer apparatus. The soaking method does not require a return line but means should be provided, such as a rubber hose, to carry the spill-over from the vent to a safe location, and the pump discharge should be connected to the lowermost inlet to the unit. Depending upon the volume of the liquid to be handled, the pump discharge and the return or vent spill-over lines, as the case may be, are usually of 40 to 75 mm pipe size.

The filling or circulating pump should not be fitted with any bronze or brass parts and it should have the capacity to deliver, in not over 1 hour, at 690 kPa (7 kg/cm^2), a volume of liquid equal to the capacity of the vessel to be cleaned. For convenience in handling and mixing the solution, the sump tank should be large enough to hold about 5 percent of the total volume of liquid required to fill completely the vessel to be

cleaned. An adequate supply of clean water and steam for heating the solvent to the desired temperature should be provided. Provision should be made for adding the inhibited acid to the suction side of the filling or circulation pump.

7.3 Cleaning Solutions — To determine the amount of solvent required, an estimate should be made of the capacity of the vessel to be cleaned, adding 2 percent to allow for wastage and other contingencies. Commercial acid suitable for the deposit to be removed should be procured in the quantity required to make, when mixed with water, a solvent of the amount and acid strength desired.

7.3.1 An inhibitor suitable for the acid to be used should be procured. In obtaining the manufacturer's recommendation for the amount of inhibitor to be used, care should be taken to make certain that this recommendation is based on full knowledge of the acid strength of the solvent and purpose of use, to avoid any possibility of a higher corrosion rate than can be tolerated when cleaning a boiler. The amount of inhibitor specified for each carboy of acid should be completely dissolved or suspended in about 3·5 litres of acid removed from the carboy, taking care not to let the solution heat. After pouring the mixture of inhibitor and acid back into the carboy, the contents should be thoroughly mixed (this can be done for small boilers). Inhibitor efficiency should also be tested during circulation by immersing a degreased nail or steel wool ball in a sample of the liquid to see evolution of hydrogen.

7.3.2 In preparation for the neutralizing process, commercial soda ash should be provided to make up a solution sufficient to fill the unit completely in the proportion of 1 kg of soda ash to 100 litres of water. This will produce a solution strength of about 1 percent.

7.3.3 If the operator retains an organization specializing in acid cleaning of heat transfer equipment, the latter ordinarily determines and supplies the type of cleaning solvent and handling equipment best suited for the conditions.

8. CIRCULATION METHOD

8.0 In modern chemical cleaning practice, circulation method is more common than soaking method as the chemical cleaning of boilers can almost go for completion in circulation method and also as it is more economical because of the lesser amount of chemicals used. In this standard, therefore, only circulation method has been described in detail. Circulation method is followed in the following two types of chemical cleaning of boilers:

a) Preoperational cleaning of boilers, that is, cleaning done before a new boiler is commissioned (**8.2**); and

b) Postoperational cleaning of boilers, that is, cleaning done after the boiler has been in service for a certain period of time (**8.1**).

8.1 Postoperational Chemical Cleaning of Boilers

8.1.1 *Chemical Cleaning Frequency* — The following recommendations are made for estimating chemical cleaning frequeney:

a) Maintain a 'need for chemical cleaning' log for each high-pressure boiler. This log should include a record of all known deviations from cycle chemistry. The following should be included:

   1) the number of outages,

   2) method of standby protection,

   3) length of operation when feed water quality limits were not maintained,

   4) length of operation when boiler water quality and treatment limits were not maintained,

   5) number of suspected and confirmed condenser leaks, and

   6) other contamination of the cycle, such as evaporator carryover or demineralizer maloperation.

b) Perform sufficient routine chemical analyses to adequately define the normal cycle chemistry environment. Metal ion (Fe, Cu and Ni) determinations should be included in the analysis programme. Measurements of these constituents should be made twice weekly on the feed water during normal operation and more frequently during start-up to establish boiler crud loading.

c) Establish a cleanliness history for all high-pressure boilers based on periodic reviews of the 'need for chemical cleaning' log and cycle chemical analysis results. Verify decisions regarding internal cleanliness of all boilers through an annual tube sample analysis programme.

d) Do not operate any high-pressure boiler longer than 4 years between chemical cleanings. This frequency is necessary to assure long-term boiler integrity. Even though the steam generator may have operated without cycle chemistry deviation, chemical cleaning should be considered and utilized on a 4-year basis as a proper maintenance tool.

8.1.2 *Method of Tube Sampling* — Selecting the location of tube samples from a boiler for deposit measurement is very important since deposits may vary appreciably in different parts of the boiler. Tube samples

should be removed from locations which generally represent areas of heaviest corrosion product deposition. Sampling in a band starting at the centre line of the uppermost burner and extending up the furnace about 3 000 mm is recommended. Operating experience in specific units, however, will often determine other problem areas that should be looked at, such as arch tubes in natural-circulation boiler or other horizontal or low-sloped circuits.

8.1.2.1 Once the tube sample location is selected, the actual method of tube removal is important to assure a useful sample. Three adjacent tubes should be removed for comparison analysis. Cut-off wheels and cutting torches produce slag and fillings, which adhere to the inside tube surface near the cutting point. The sample, therefore, should be at least 600 mm in length to negate this contaminating effect. After the tube has been removed, it should be sealed on the ends and sent to the analysing laboratory for measurement of deposit amount and composition of deposits.

8.1.3 *Relationship of Deposit Quantity to Unit Cleanliness* — The amount of deposition on clean surfaces is usually less than 15 mg/cm^2 while on moderately dirty surfaces it may be 15 to 40 mg/cm^2 and on very dirty surfaces, more than 40 mg/cm^2. Chemical cleaning should be done whenever deposits are more than 40 mg/cm^2.

8.1.4 *Location of Temporary Equipment* — The location of the temporary equipment may be decided based on the following considerations:

a) The dissolving tank should be elevated to suitable height to provide the net positive suction head (NPSH) specified for the acid circulating pumps. A suitable location in the boiler floor area can preferably be looked into. Otherwise the tank has to be supported externally on temporary structures.

b) Sufficient care should be taken to locate the dissolving tank at a place where damage to the nearby equipment due to the possible spillage of chemical solutions from the tank by overflowing is avoided.

c) Sufficient space should be provided between the pumps to have enough access for safe operation.

d) Circulating pumps and starters should be separated at a safe distance to have safety and ease of operation.

e) It is preferable to locate the pumps nearer to the waste disposal point such as main station drain, etc.

f) The main controlling valves, such as recirculating valve and valves for make-up water, steam, system return, and system drain, should be located near the circulating pump at conveniently accessible location for effective control of the process.

8.1.5 *Neutralizing Arrangement* — All the chemical effluents have to be neutralized suitably before leaving them off to sewerage. Special care has to be taken to neutralize the spent acid after the completion of pickling operation effectively if the effluents are discharged to a canal or river meant for agricultural or domestic consumption. The spent acid may be effectively neutralized with burnt lime in an open concrete type storage tank of around 20 m^3 capacity. The tank may be located outside boiler area from where it can be connected by open channel to the main stream. The tank shall have two or three compartments to provide sufficient retention time for effective neutralization. After the initial neutralization in the tank the effluents can be further neutralized in the open channel by progressive addition of neutralizing chemicals at stages during the passage to the main stream. The effluents, after neutralization, shall be diluted with enormous quantity of water taken through a fire-fighting point or from some other source. If a suitable alternative neutralizing arrangement exists, say in the water-treatment plant, the same can be made use of. Hydrazine may be oxidized with bleaching powder.

8.1.6 *Nitrogen Capping* — Nitrogen capping is adopted to protect the freshly cleaned metal surfaces after pickling from being corroded due to exposure to atmospheric air during the subsequent stages of acid cleaning operations.

8.1.6.1 Required number of cylinders of suitable capacity are connected to the bus which in turn is connected to the drum air vents through a stop valve and a non-return valve. In case smaller cylinders are envisaged for the capping, two manifold systems to accommodate 15 cylinders each, connected through two heavy duty regulators, may be employed.

8.1.6.2 Nitrogen capping should be done during each dumping for all operations following pickling. The stop valve in the nitrogen line and the air vent valve nearer the drum are kept open while the other air vent valve on the drum is kept closed. All other air vents in the system are kept closed to prevent ingress of air into the system. As the dumping of the system starts nitrogen enters through the non-return valve into the system and offers effective blanketing. When the system is completely drained and filled with nitrogen, the stop valve in the nitrogen line shall be closed. The pressure of nitrogen in the system is to be maintained slightly above atmospheric pressure, say about 35 to 70 kPa (0·35 to 0·70 kg/cm^2). When the system is getting filled for next operation, the nitrogen in the system is vented through the air vents. The non-return valve in the nitrogen admission line prevents entry of water or chemical solution to the nitrogen piping system.

8.1.6.3 Nitrogen capping may be terminated after first stage passivation.

8.1.7 *Connecting Point Details*

8.1.7.1 The temporary equipment and connected pipelines and valves shall be erected in accordance with the final layout prepared by the customer/consultant depending upon site conditions.

8.1.7.2 Economizer inlet connection may be given at the main isolating valve towards economizer after the non-return valve in the main feed control point. The connection may be given at the valve after removing the valve wedge and bonnet assembly. However, it is preferable not to erect the valve to accommodate temporary connections.

8.1.7.3 The nitrogen capping connection should be given at the drum air vents.

8.1.7.4 The degasifying header connections in the drum are to be made.

8.1.7.5 Bottom ring header connections have to be made through hand-holes.

8.1.7.6 Superheater should be plugged before commencement of pickling operation.

8.1.7.7 Downcomers should be orified suitably for the cleaning process.

8.1.7.8 Hydrostatic plugs may be assembled into the safety valves after protecting the seats of the valve with laquer or plugging the safety valve connections from inside the drum.

8.1.7.9 The vent lines from economizer and drum are to be led to a tank of 1 m^3 capacity located at drum level.

8.1.7.10 The drains from economizer, and blowdown header and ring header are to be connected to the neutralizing pit.

8.1.7.11 Whenever the economizer recirculation lines are present, the non-return valve flaps connecting the downcomers to economizer inlet header may be removed and also the isolation valve may be kept open during pickling and subsequent operations.

8.1.7.12 For filling the superheater, a line from circulating pump discharge header should be connected to superheater header drain.

8.1.7.13 For intermittent pressurizing during pickling, if required, a separate line through a pressurizing pump may also be connected to the superheater header drain at a convenient location.

8.1.8 *General Details*

8.1.8.1 Provision of sampling points should be made at the pump discharge and return line to the mixing tank. Draining facilities should be adequate to drain the entire system preferably within one hour.

8.1.8.2 The chemical cleaning should commence at a time to be mutually decided upon by the parties concerned. However it is prudent to commence chemical cleaning operations at a time when the overall commissioning programme of the boiler is geared up to go on without any interruption. Completion of chemical cleaning at a premature stage will not realize the full advantage of the process.

8.1.8.3 All the weldings in the temporary pipelines should be done with high-pressure welders and should be of radiographic quality.

8.1.8.4 The pumps should be thoroughly serviced before commencing the operation and should be given a trial run first.

8.1.8.5 All the temporary valves should be serviced before commencing for easy operation.

8.1.8.6 Gland packing shall be of pure polytetrafluoroethylene.

8.1.8.7 During chemical cleaning, especially during dosing of chemicals, the adherence to safety regulations for handling acids, caustics, corrosive chemicals, etc, shall be strictly observed. The persons carrying out the chemical cleaning operation should be equipped with goggles, rubber gloves, boots, etc.

8.1.8.8 Adequate lighting facilities should be provided in the boiler area.

8.1.8.9 Sufficient number of telephones should be installed at strategic locations for effective control of the process.

8.1.8.10 An established laboratory facility should be made available along with enough trained chemists to carry out the analysis during the process effectively on the spot.

8.1.8.11 A suitable alternate power source for at least one or two acid circulating pumps should be made available to meet out any emergency in the event of power failure.

8.1.8.12 The chemical cleaning should be done under close technical and chemical supervision.

8.1.8.13 Before commencing alkali boil-out, all major pipelines in the pre-boiler system should preferably be mass flushed and cleaned.

8.1.8.14 Before commencing alkali boil out feed pump should be trial run and made ready for operation.

8.1.8.15 Before commencing the process, sufficient amount of demineralized water storage with filling to dissolving tank at the rate of about 100 t/h should be ensured.

8.1.8.16 For the complete cleaning process sufficient amount of heating steam at about 1 MN/m^2 and 250°C should be made available.

8.1.9 *Post Operational Chemical Cleaning of Boiler with Economizer But Without Super Heater* — The technological procedure for chemical cleaning is as given in **8.1.9.1** to **8.1.9.12**.

8.1.9.1 *Water washing* — Before commencing alkali boil-out, the boiler is flushed with water as follows:

The boiler is filled through economizer, using boiler filling pump up to the top of the drum, taking care that the water does not enter the superheater. After filling, the water is drained in the shortest possible time by opening all the drain valves. This process is repeated two or three times till the water collected from drains is reasonably clear.

8.1.9.2 *Alkali boil-out* — The alkali boil-out operation is performed by actual firing of the boiler at half its working pressure, or at 3·9 MN/m² (40 kg/cm²) gauge when boiler working pressures are above 7·8 MN/m² (80 kg/cm²), with 2 000 mg/l of trisodium phosphate. This pressure as the case may be is maintained for a maximum of 48 hours. Trisodium phosphate may be dissolved in buckets and directly charged into the drum through drum manhole. Before charging the chemical, the boiler should be filled up to the bottom of the drum. After addition of chemicals, before light up, the boiler is filled up to normal operating level.

The boiler is lighted up in accordance with standard instructions and pressure is raised gradually to half the boiler working pressure or 3·9 MN/m² (40 kg/cm²) as the case may be. Hold this pressure for 48 hours with hourly blowdowns through individual blowdown valves lasting one minute each to remove substantial quantities of impurities collected in the water wall headers. During the operation the feeding rate is maintained to keep normal level in the drum. Samples are taken every two hours and analysis carried out for *p*H, alkalinity and conductivity. Care should be taken to keep the furnace outlet flue gas temperature of superheater/reheater within limits. After 48 hours of boil-out, the system is drained hot completely at permissible pressure. When the boiler water alkalinity and phosphate concentration drops to half of their original values additional chemicals may be added to raise alkalinity.

8.1.9.3 *Rinsing after alkali boil-out* — The rinsing after boil-out is done, after the boiler is considerably cooled, as described for water washing till the system is free from alkali solution, that is, *p*H of the effluent is equal to that of the water used for rinsing. The system is then made ready for acid pickling after superheater is filled and plugged, drum internals removed and downcomer and water wall downcomer orifice plate fixed, and nitrogen blanketing connection is made.

8.1.9.4 *Filling of superheater with hydrazine water* — Hydrazine (200 mg/l) and ammonia (to raise *p*H to 9·6) are added to demineralized water in dissolving tank and slowly pumped to the superheater. When the *p*H of water rises to 9·6 at connecting tubes from superheater in drum, the connecting tubes are plugged with special temporary accessories. The superheater is kept under pressure (say 0·5 MN/m^2) throughout the cleaning process. Suitable arrangements have to be provided for pressuring superheater during pickling, if required.

8.1.9.5 *Acid washing* — The system is filled with demineralized (DM) water and circulation established. The temperature of the water is raised and maintained at 65°C by admitting steam to the dissolving tank. The charging procedure is as follows:

> First, inhibitor is charged full and after few minutes the hydrochloric acid and ammonium bifluoride (minimum 0·5 percent by mass) are charged simultaneously. The addition of chemicals should be completed within one hour to reach the required concentration of 4 percent. Care should be taken to see that the hydrochloric acid concentration in the cleaning solution does not increase beyond 5 percent at pump suction. The acid concentration should also not fall below 3 percent.

During acid circulation, the concentration of hydrochloric acid in the system is checked every 15 minutes, and of ferric iron every half hour. Due to chemical reactions during washing process, the concentration of hydrochloric acid decreases rapidly at first and slowly afterwards and then it steadies at a constant value. Similarly, iron value increases and attains a steady value. The constant values of hydrochloric acid and iron content denote the completion of acid washing. The system is then drained under nitrogen blanketing and the drained acid is neutralized before disposal. When the ferric iron content of the inhibited acid reaches 0·4 percent, the acid is drained and fresh charge of inhibited acid is used as ferric iron is highly corrosive for steel.

8.1.9.6 *DM rinse* — After draining the pickling solution completely, the boiler is filled with DM water and circulation established. When uniform concentrations of acid and iron are attained, in the system as indicated by analysis, it is drained under nitrogen blanketing. The *p*H of the DM rinse should not be allowed to go above 4·0 in order to prevent precipitation of iron chloride as iron hydroxide which may settle down on cleaned boiler surfaces.

8.1.9.7 *Citric acid rinse* — The system is again filled with DM water containing 0·2 percent by mass of citric acid. While filling the system with DM water add proportionate quantity of citric acid so as to give a uniform concentration of 0·2 percent. Steam may be admitted to raise the temperature to 55 to 60°C. If required ammonia may be added to bring up the *p*H of the circulating solution to 3·5—4·0. After attaining

uniform concentration throughout the system circulation is maintained for one hour and the system drained under nitrogen blanketing. This citric acid rinse may be repeated with advantage for the effective removal of dissolved iron from the system.

8.1.9.8 *DM rinses* — The system is filled with DM water. Circulation is established and maintained for one hour. After one hour of circulation the system is drained under nitrogen blanketing. During the rinsing operations the drain samples are checked for *p*H and iron content. The rinsing operations are repeated till the iron content of the solution drops down to less than 25 mg/l and its *p*H approaches that of DM water.

8.1.9.9 *Soda ash washing* — The system is filled with DM water containing 1 percent by mass of sodium carbonate. Proportionate amounts of sodium carbonate may be added while filling the system itself. After complete addition of chemicals and establishing circulation the temperature is raised to 85°C and circulation maintained for 4 to 6 hours at this temperature. The solution should be circulated through the system until the return water shows a strong alkaline reaction, as indicated by a sample turning red on addition of a few drops of phenolphthalein. The same test should be made on samples from all sampling points and vents, to make sure that the soda ash solution has reached all areas previously reached by the acid. After the circulation is discontinued and soda ash solution is lowered to the normal operating level, the unit should be fired to raise the steam pressure and to hold it for 2 hours at 0·3 to 0·6 MN/m^2. This will aid in cleaning the surfaces and will also drive off hydrogen from the metal. The unit should be vented several times during this period to get rid of the hydrogen liberated. It is absolutely essential to vent out all the absorbed hydrogen from the boiler metal to prevent any possible embrittlement of steel due to hydrogen. When hydrogen venting is over, the firing of the unit is stopped and the pressure reduced to zero. The unit is flushed with hot DM water for about 1 hour and during this period the vents are opened several times to get rid of the liberated hydrogen. Finally, the unit is drained under nitrogen blanketing.

8.1.9.10 *Passivation* — The object of passivation is to prevent corrosion since the cleaned surfaces are more susceptible to corrosion which takes place at a very fast rate. The corrosion is prevented by providing a thin layer of magnetite (first stage) which gets strengthened in the subsequent firing of the boiler (second stage). The procedure for passivation is detailed below:

a) *First stage passivation* — The system is filled with DM water and adjusted for circulation. The temperature of water is raised and maintained at 85°C by external steam heating. *p*H of DM water is raised to 9·5 by adding ammonia and hydrazine is charged to

get a concentration of 200 mg/l. The solution is left to circulate for about 20 hours at a temperature of 90 to 95°C. The hydrazine concentration is maintained at 200 mg/l throughout the operation. After 20 hours, the solution is drained hot from the system which is left to aeration by natural draft.

b) *Inspection of first stage passivation* — The drum is inspected thoroughly and all loose debris cleaned manually. Ring header hand-hole plate connections are removed and the header cleaned with water.

c) *Preparatory work for second stage passivation* — The superheater plugs and downcomer orifice plates are removed and the drum internals refitted. All the provisional equipment is dismantled and original fittings and mountings installed back.

The boiler should be prepared as for normal operation.

d) *Second stage passivation* — The chemicals namely ammonia liquor, for raising the *p*H to 9·6, and hydrazine, to get a concentration of 200 mg/l, are charged in the chemical dosing tank. The boiler is filled with feed pump to normal operating level in drum and lighted up with one or two oil burners. The steam pressure in drum is gradually raised to 4 MPa or half the working pressure, whichever is lower, and maintained at that value. Water samples are taken from feed line to find out hydrazine concentration and *p*H value. The boiler is kept in service for 24 hours, maintaining the hydrazine concentration not less than 25 mg/l in feed water. If the header drains are to be operated the fire should be killed.

At the end of the process, the boiler is boxed up and allowed for natural cooling. At 100 or 200 kPa or the pressure recommended by the boiler manufacturer, the superheater air vents are opened, and at about 90°C (drum temperature), the water in the system is drained and the system is left to aeration by natural draft.

The second-stage passivation marks the end of the chemical cleaning process. It is advisable to put the chemically clean boiler into service within a period of seven days.

If it has not been possible to light up the boiler again within 7 days, next fill of 200 mg/l of hydrazine and ammonia for *p*H value 9·6 should be given and the system boxed up completely with an intermittent checking for the values of *p*H and hydrazine content, which should be made up by an extra dosing of hydrazine.

NOTE — The time gap between the first and second stage inspections should be not more than 7 days.

8.1.9.11 *Boiler deposits with copper up to 5 percent* — When boiler deposits contain copper up to 5 percent, then copper is also removed along with iron in a single-step cleaning. But to prevent the dissolved copper plating the boiler drums and tubes, the dissolved copper is complexed with thiourea and removed.

8.1.9.12 *Boiler deposits with copper more than five percent* — When copper content in the deposits is known to be higher than 5 percent, a removal step is first carried out before acid cleaning. In this process, trisodium ammonium citrate solution is used at 50°C and at a *p*H value of 10·0 with the addition of ammonia together with an oxidising agent such as sodium bromate or sodium nitrite.

8.1.10 *Post Operational Chemical Cleaning of Boilers (with Superheater Included)* — When superheater is included in the boiler chemical cleaning, it is essential to check first the metallurgy of superheater element. If it is made of low chromium steel (chromium less than 5 percent) the same inhibited hydrochloric acid may be used for chemical cleaning of superheaters. But, if superheater is made of high chromium steel or stainless steel then the cleaning should be done with inhibited 3 percent citric acid to dissolve the scales in the superheater but never inhibited hydrochloric acid as it would initiate chloride stress corrosion cracking. When superheater has to be chemically cleaned, it is normally done after chemical cleaning of boiler drum and tubes as detailed under **8.1.9**. This is done to prevent the major portion of deposits removed from boiler depositing in the superheater tubes during circulation.

8.2 Preoperational Chemical Cleaning of Boilers

8.2.1 *Cleaning of Boilers Without Superheater* — The same procedure as detailed for postoperational cleaning of boilers without superheater (*see* **8.1.9**) is followed.

8.2.2 *Cleaning of Boilers with Superheater* — The same procedure as detailed for postoperational cleaning of boilers with superheater (*see* **8.1.10**) is followed. During flushing operation of superheater velocity of water should be about 1·5 m/s.

9. PRECAUTIONS AND MEASURE OF RESULTS

9.1 Cleaning should not be considered a substitute for proper water treatment. The rigid control maintained in well operated plants to achieve the best possible water treatment should not be relaxed because of the relative ease of cleaning by the chemical method. Chemical cleaning is ordinarily not necessary more often than once in three years. Intervals between cleanings should be extended or reduced as conditions warrant. This is essential since each time a unit is chemically cleaned the metal surfaces are depacified or stripped of their protective oxide coating, and in addition, a slight amount of parent metal is dissolved because none of the known inhibitors is completely effective.

9.1.1 In handling acids, the recognized precautions of using goggles, rubber gloves and rubber aprons should be observed. Acid should be poured slowly into water; never should water be poured into acid.

9.1.2 Acid cleaning removes mill scale and products of oxidation. A thorough visual inspection should be made after cleaning to determine whether any parts need replacement.

9.1.3 Undrainable pressure parts, such as pendent-type superheater tube elements, require special attention to make certain that they are cleared of all acid solvent and loose deposits. Usually each individual element should be separately flushed through a tight fitting hose connection, using a similar arrangement at the discharge end to avoid spilling of the flushing discharge into other elements. A mixture of compressed air and water as the flushing medium has also been used effectively.

9.1.4 The chloride content of the citric acid, particularly used for superheater cleaning, should not be more than 0·1 mg/l as Cl in its 0·2 percent (*m/m*) solution.

9.1.5 In case of entry of chemicals to superheater, it should be back-flushed properly to the drum and care should be taken for proper steam-blowing at later stages before the turbine is taken into circuit. This shall be strictly followed both during preoperational and post-operational cleaning processes.

9.1.6 In riveted joints the solvent may enter seams, and damage can occur without being detected. Therefore, chemical cleaning of riveted drums is usually avoided. This applies particularly to drums calked on the outside.

9.1.7 The possibility of hydrogen gas being generated during acid cleaning of internal heating surfaces should never be overlooked. The quantity of hydrogen released is a measure of the ineffectiveness of the inhibitor. A portion of the hydrogen is absorbed by the metal, while the remainder forms part of the atmosphere in the vessel. The portion absorbed by the metal is liberated gradually, the time required decreasing as the temperature of the metal increases. While the metal wastage indicated by its generation may be inconsequential, the hydrogen is a source of two potentially dangerous conditions, namely, embrittlement of the steel and an explosive atmosphere in the vessel.

9.1.8 On completion of acid cleaning, because of the possibility of hydrogen embrittlement, the steel should not be cold-worked (tubes rolled, flanges tightened, seams calked, etc) in any manner for at least four or five days until the pressure boiling out has been completed or the boiler has been brought up to steam pressure in the normal manner.

9.1.9 In opening a unit for inspection after acid cleaning, the possibility of an explosion owing to the presence of free hydrogen in the atmosphere within the unit should be recognized. There have been cases of explosion of hydrogen atmosphere in boilers after acid cleaning where fatal injuries have resulted. At atmospheric pressure and ordinary temperatures, the values for hydrogen content indicated for the inflammability of a mixture of hydrogen in air range from 4 percent to 75 percent. The ignition temperature of hydrogen in air, at atmospheric pressure, is between 580 and 590°C.

9.1.10 Because of the possibility of an explosion, the following precautions should be observed.

9.1.10.1 After acid cleaning, no one should enter the unit or get in the way of any openings to the pressure parts unless the atmosphere within has been cleared of explosive gases. Until this has been done, open flames, flashlights, any other lighting equipment, or anything that might produce a spark of any kind should not be used within or near the openings of pressure parts.

9.1.11 The following steps may be taken to clear the atmosphere of explosive gases within the pressure parts.

9.1.11.1 Following an acid cleaning, the unit should be thoroughly flushed with warm water to a positive overflow from the topmost vent to besides other, make certain that all the atmosphere within has been displaced. To warm the unit and thus accelerate the libration of hydrogen absorbed in the metal, the temperature of the water should be held as nearly as possible at 100°C by light firing or other means.

9.1.11.2 After the unit has been opened, air eductors, placed at locations such as open drum manholes, should be used to circulate air through the unit for removal of hydrogen being liberated from the metal. This should be continued until analysis by a Burrell or other suitable instrument shows that the air within is free of explosive gases.

9.2 Measure of Results — Determination of the type and approximate amount of different kinds of scale and impurities removed from a unit at a cleaning will be possible if, at periodic intervals during the operation, the spent cleaning solvent and the water and solid matter ejected are properly sampled and analysed and a record is kept of the total amount involved. This information serves as a valuable guide for further boiler-water treatment and the determination of the required frequency of cleaning.

9.2.1 Finally, while chemical cleaning with inhibited acid is in progress, a cut piece sample of boiler tube should be kept hung in the boiler drum throughout the acid cleaning period to see the effect of the inhibited acid on the boiler tube cleaning. At the same time a well cleaned and weighed small cut piece of boiler tube should also be kept hung in the same boiler drum during the acid cleaning operation and weighed again after the completion of acid cleaning operation. The loss in mass would denote the corrosion on the boiler due to the use of inhibited acid. A typical corrossion rate will be 2·5 g/m^2 hour.

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